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EFFECT OF MOISTURE ON SURFACE FLAMMABILITY OF COATED AND UNCOATED CELLULOSIC MATERIALS

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U.S. DEPARTMENT OF COMMERCE
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Effect of Moisture on Surface Flammability of
Coated and Uncoated Cellulosic Materials

T. G. Lee, J. J. Loftus, D. Gross

ABSTRACT

A method is described for the rapid in situ determination of the "thermal inertia for surface heating" ($k\rho c$ product) of materials using a sensitive infrared detector. Experimental measurements are given of the effect of moisture content on the thermal inertia and on the surface flammability of selected cellulosic materials conditioned to equilibrium at relative humidities ranging from 0 to 99 percent. It is shown that (1) the thermal inertia of uncoated hardboard and fiberboard material can be represented as a linear function of its moisture content, (2) that if the appropriate thermal properties are used, the flame-spread factor is inversely proportional to thermal inertia, and (3) the unbroken surface film of coated materials results in a lower ignition sensitivity than that predicted on the basis of thermal inertia.

1. Introduction

The flame-spread index resulting from the performance of a standardized flame-spread test on a material [1, 2] has been shown to be composed of two multiplicative components: (a) a flame-spread factor representing the ignition sensitivity of the material, and (b) a heat evolution factor representing the maximum rate of heat generation [3]. A recent study of surface flame propagation on cellulosic materials showed that, except for very thin veneers, the flame spread factor was inversely proportional to the $k\rho c$ product, the "thermal inertia for surface heating" [4]. Here k , ρ , and c are the thermal conductivity, density and heat capacity, respectively. These results appeared to support a simple concept for the spread of flame on the surfaces of cellulosic materials exposed to thermal radiation, namely that flame propagation consists of progressive ignition of the solid when a characteristic surface temperature is reached.

The correlation between the flame spread factor and the $k\rho c$ product was based upon a measured value for ρ and individual values of k and c obtained from the handbook. The present paper describes a simple method for determining the thermal inertia of a material from a recording of its surface temperature history during surface irradiation. This technique was originally used for measurement of rapid changes of human skin temperature [5] and is particularly attractive for in situ measurements on building materials, including wood and other inhomogeneous materials.

Because of the strong influence which moisture content has upon surface flammability, a study was made of the effect of moisture content on (a) the thermal inertia, and (b) the flame spread behavior of selected cellulosic materials. The main objective of this study was to evaluate the overall effect of moisture content and to illustrate the importance of the thermal inertia property on surface flammability without consideration of the effects of diffusion and phase change of moisture.

2. Theory

The surface temperature rise θ_s of a homogeneous semi-infinite solid which is inert, opaque and totally absorbing, subjected to constant irradiance I on the surface and losing heat by Newtonian cooling at the surface is given by the relation [6]:

$$\theta_s = \frac{I}{H} [1 - e^{-b^2} \operatorname{erfc} b] \quad (1)$$

where $b = H \sqrt{\frac{t}{k\rho c}}$, erfc is the complementary error function,

H is the surface heat loss coefficient,

t is time, and k , ρ and c are the thermal conductivity,

density and heat capacity, respectively. It is assumed that heat flow is one-dimensional and that the thermal properties, which may represent effective values for moist solids, are independent of temperature.

For sufficiently short times or for low values of the heat transfer coefficient (see Appendix 1), an approximation to the surface temperature rise is the more common expression:

$$\theta_s = 2I \sqrt{\frac{t}{\pi k\rho c}} \quad (2)$$

To account for specimen absorptivity less than unity, it is only necessary to prepare all surfaces in a similar manner and to consider the "absorbed" irradiance $I = \alpha I_0$, where α is surface absorptivity and I_0 is the incident surface irradiance. While the assumption of opacity is satisfactory for many materials, natural wood is considered partly diathermanous. It has been found, however, that blackened wood surfaces are essentially opaque [7].

Equation (2) may be rearranged to obtain:

$$k\rho c = \frac{4 I^2 t}{\pi \theta_s^2} \quad (3a)$$

It is sometimes desirable to supply incident radiation periodically in order to eliminate energy reflected directly from the irradiated surface to the detector when there is a spectral bandwidth overlap. In this case, a time factor f , which represents the ratio of irradiation time to total time, must be introduced. Using this factor, a simplified expression may be written for the surface temperature rise (see Appendix 2) of an inert, opaque semi-infinite solid subject to periodic application of a constant effective irradiance with no heat losses:

$$k\rho c = \frac{4 f^2 I^2 t}{\pi \theta_s^2} \quad (3b)$$

Determination of k_{pc} , the thermal inertia for surface heating, simply requires (a) a sufficiently rapid recording of the temperature rise history of a blackened surface and (b) knowledge of the magnitude of the effective irradiance, and its application period, when appropriate. For measurement times exceeding those listed in Table 4, knowledge of the surface heat loss coefficient is also required, and the value of k_{pc} is then obtained from equation (1) by using the plot of $(1 - e^{b^2} \operatorname{erfc} b)$ versus b .

3. Experimental Method

3.1 Surface Temperature

A non-contact radiometric technique was used successfully by Hardy and his associates [5, 8] for the rapid measurement of the surface temperature of the human skin and for determination of its thermal properties. This technique involves comparison of the infrared radiation from the object with that from a blackbody source of known temperature.

The experimental arrangement for measuring surface temperatures used in the present study is shown schematically in Fig. 1. The source of radiant heat was a 300 watt projection lamp mounted in a ventilated housing. A line-voltage regulator and autotransformer supplied power to the lamp and permitted close control of the radiant intensity. The radiation from the lamp passed through a 2 micron low pass filter and impinged on a circular area, 8 cm in diameter of the specimen. The irradiation, which was typically of the order of 0.02 watts per square centimeter (w/cm^2), was found to be uniform to within ± 3 percent on a circular area 2 cm in diameter with a gradual fall off to the edges of the irradiated area. An opaque shutter mounted between the lamp and the specimen was used to control exposure duration.

The radiometer used for indicating temperature changes was a commercial infrared "radiation thermometer" containing a thermistor bolometer detector with high sensitivity through the spectral bandwidth of the instrument. The instrument employed germanium lens optics giving a 3-degree field of view and a spectral response from 2 to 22 microns. The system response time was 50 milliseconds and the sensitivity under the conditions of use was on the order of 0.1°C . The radiometer was focused on a circular area of about 1.2 cm in diameter in the center of the irradiated area where the effect of lateral heat conduction was negligibly small.

In operation, a motor-driven reflective chopper wheel, within the radiometer, interrupted radiation from the target and reflected radiation from a controlled internal blackbody on to the detector at a rate of 90 cycles per second. Thus, a 90 cps signal was generated whose amplitude was proportional to the difference in radiance between the target and the internal source. This signal was amplified, rectified and recorded.

The radiometer was calibrated at the start and end of each day by substituting for the specimen a large copper block with a black-coated cylindrical cavity. The temperature of the block was raised by means of an imbedded electrical heater and monitored by means of a precision thermometer also imbedded in the block. A series of measurements of detector output readings and copper block temperatures was made over the operating temperature range (20° to 30°C). Since the same flat black coating was used on the copper block cavity as on the test specimens, the calibration provided a means for measuring the surface temperature of the specimen.

For applications in which energy reflected from the irradiated surface to the detector may introduce a spurious response (i.e., when the spectral bandpass of the detector overlaps the wavelength region of the incident radiation), a second chopper wheel can be placed so as to periodically interrupt radiation incident on the specimen. Exact out-of-phase synchronizaton with the similarly interrupted target radiation would thus avoid the receipt of any reflected energy. A synchronous chopping arrangement of this sort was employed for measuring the thermal inertia of human skin during exposure to far infrared radiation [8].

3.2 Flame Spread

The apparatus and experimental procedure for performing a standardized flame-spread test has been described in detail [2, 3]. The test requires a 6- by 18-in. specimen facing and inclined 30 degrees to a vertically-mounted, gas-fired radiant panel. Ignition was initiated at the upper edge of the test specimen and observations were made of the progress of the flame front down the specimen surface as well as of the temperature rise of thermocouples in a stack supported above the specimen. The test duration was 15 minutes, or until sustained flame propagated downward the entire 18-in. length of the specimen, whichever time was less. The flame-spread index I_s was computed as the product of the flame-spread factor F_s and the heat evolution Q .

$$\text{or } I_s = F_s Q \quad (4)$$

$$\text{where } F_s = 1 + \frac{1}{t_3} + \frac{1}{t_6 - t_3} + \frac{1}{t_9 - t_6} + \frac{1}{t_{12} - t_9} + \frac{1}{t_{15} - t_{12}} \quad (5)$$

The symbols $t_3 \dots t_{15}$ correspond to the times in minutes from specimen exposure until arrival of the flame front at a position 3 . . . 15 inches, respectively, along the length of the specimen. The heat evolution Q is proportional to the observed maximum temperature rise of the stack thermocouples.

3.3 Specimen Preparation

Although values of thermal inertia of other types of cellulosic materials have been determined by this method, the present study on the effect of moisture was confined to only two materials - cellulose fiberboard and tempered hardboard. These were chosen for the initial study in order to minimize the effect of structure inhomogeneity which is characteristic of natural woods. Table 1 lists the thickness, density, humidity condition and corresponding equilibrium moisture content for each specimen. The relative humidity-equilibrium moisture content relationships are shown graphically in Fig. 2.

A test specimen was prepared by applying a flat black enamel to a circular area of 7.5 cm diameter near its center. This coating was spray-applied and contained approximately 8% pigment, principally carbon black, and 8% non-volatile modified styrene vehicle. The normal paint coating, when dry, weighed 0.002 g/cm^2 and this produced a surface of high absorptivity (>0.94) to visible and near-infrared radiation. The 6 by 18 in. specimen was then dried in an oven at 105°C for 24 hrs, allowed to cool in a desiccator, weighed, and then placed in a series of controlled humidity chamber to reach constant weight.

The humidity chambers (Table 2) consisted of (a) moisture-tight drums containing either calcium chloride or various saturated salt solutions [9] for maintaining a series of specified relative humidities, (b) a commercial room air conditioning system for maintaining 50 ± 5 percent relative humidity (rh) and (c) a commercial controlled humidity chamber especially suitable for controlling humidity in the 80 to 100 percent rh range. The relative humidity in all chambers was monitored by means of commercial moisture-sensitive resistance elements calibrated to ± 2 percent rh.

Specimens were transferred in polyethylene bags from their humidity chambers for weighing and thermal inertia determination. Prior to removal from the bag, specimens were permitted to adjust to the temperature of the laboratory ambient air, when necessary. Thermal inertia determinations were then made as quickly as possible. Although the effect of variations in laboratory ambient humidity (which ranged from 35 to 50 percent rh) was not studied, some exchange of moisture between specimen and ambient atmosphere would be expected.

After the thermal inertia determination, the specimen was returned to its controlled humidity chamber for reconditioning prior to a flame-spread test. Additional specimens were conditioned and tested for surface flammability only to provide sufficient flame-spread replication data.

4. Results and Discussion

Fig. 3 illustrates typical surface temperature-time records for fiberboard and tempered hardboard of various moisture contents. When plotted as a function of \sqrt{t} , as shown in Fig. 4, the data appear as straight lines, and the initial slopes may be used to calculate values of kpc using equation (3a).

For times exceeding those listed in Table 4, equation (1) was used to evaluate kpc. The surface heat transfer coefficient was calculated from the following equation:

$$H = \frac{\sigma \epsilon (T_s^4 - T_a^4) + h (T_s - T_a)}{T_s - T_a} \quad (6)$$

Where T_s = surface temperature $^{\circ}\text{K}$

T_a = ambient temperature, $^{\circ}\text{K}$

σ = Stefan-Boltzmann constant, $5.67 \times 10^{-12} \text{ w/cm}^2 \text{ } ^{\circ}\text{K}^4$,

ϵ = surface emissivity, assumed equal to 1,

h = convective cooling coefficient.

For typical values of $T_s = 300^{\circ}\text{K}(27^{\circ}\text{C})$, $T_a = 296^{\circ}\text{K}(23^{\circ}\text{C})$ and $h = 1.8 \times 10^{-4} \text{ w/cm}^2 \text{ } ^{\circ}\text{K}$ (for laminar conditions on a vertical plane, surface with a small irradiated area), the value for H is $8.4 \times 10^{-4} \text{ w/cm}^2 \text{ } ^{\circ}\text{K}$.

Because of uncertainties associated with surface emissivity, absorptivity and diathermancy, all specimens for thermal inertia determination were painted with a flat black enamel which provided a uniformly high absorptivity and opacity. However, the actual value of the absorptivity is not necessary since a measurement was made of the effective or absorbed, rather than incident, irradiance using selected specimens of known thermal properties. The calculation of the effective I was based upon temperature-time records and independently-determined values of k , ρ and c on a variety of materials, using equation (1). Results of these measurements are given in Table 3. All subsequent computations of kpc were based on the average effective I value of 0.018 w/cm^2 .

Linear relations between moisture content and the computed values of kpc based on the present method for fiberboard and hardboard are indicated by the data plotted in Fig. 5. The dotted portion of the line shows some deviation from linearity. Natural woods of intermediate densities would be expected to have values of kpc between the two curves. The linear relationship as well as numerical values are in agreement with the findings of a previous literature survey of available data which was based on individual measurements of k , ρ and c [10].

The computed k_{pc} values take into account changes in each property due to moisture. Whereas specific heat and thermal conductivity both increase with increasing moisture content, the swelling of common woods due to moisture results in a decrease in density. This was particularly noticeable for hardboard at high moisture contents (see Table 1).

It may be noted that the presence of a thin factory-applied white paint coating on the thermal insulation fiberboard which conformed to Fed. Spec. LLL-I-535, Class D, had no appreciable effect upon the computed values of k_{pc} .

The measured flame spread factors for hardboard and for painted and unpainted fiberboard are shown in Fig. 6 as a function of the corresponding thermal inertia values. The flame spread factor is seen to be inversely proportional to thermal inertia. The plotted data include measurements over the entire range of moisture content.

A relation of this type is in accord with the concept that a characteristic temperature at the surface must be reached prior to ignition. A high k_{pc} value, for example, would delay the attainment of the characteristic temperature and thus lower the flame spread factor. In a previous study [4], flame spread factors for a wide variety of thick cellulosic materials conditioned to equilibrium at 50 percent relative humidity only, were also found to be inversely proportional to the k_{pc} values. However, the thermal conductivity and specific heat values used were "dry" values obtained from handbook sources and were not adjusted for the appropriate moisture contents. When this adjustment is made, the previous data can be represented by the equation:

$$F_s = 4 + \frac{.005}{k_{pc}} \quad (7)$$

which is also shown in Fig. 6 for comparison. Though the flame spread factors for hardboards appears to deviate somewhat from equation (7), they are actually within the data scattering on which the equation was based.

The flame spread factors for painted fiberboard were significantly different from the mean values represented by equation (7), even when the appropriate k_{pc} values were used. For flame propagation on surface-coated cellulosic materials, therefore, it appears that a higher surface temperature had to be attained in order to permit heat penetration and rupture of the coating (approximately 0.002 cm thick) and subsequent release of combustible vapors.

Figure 7 shows the measured flame spread index, $I_s = F_s Q$, as a function of corresponding computed values of thermal inertia for the three types of materials with various moisture contents. Since the heat release rate Q depends on the thickness as well as the density of the cellulosic specimens, a single continuous function is not expected to cover both the hardboard and fiberboard which do not have comparable density or thickness. But, for a given type of specimen, the correlation between I_s and kpc appears to be reasonable. Accordingly, the effect of moisture on the flame spread index may be interpreted in terms of the thermal properties of the materials.

The principal sources of error involved in the kpc determinations for this study were:

- (a) the uncertainties in the measurements of the surface temperature rise and the effective irradiance,
- (b) the modification of the surface conditions by the application of a flat black enamel, and
- (c) the use of the approximate equation (2) at long measurement times (or high values of the heat transfer coefficient).

The coefficient of variation of the kpc computations was approximately 3 percent based on fifteen sets of duplicate determinations. The overall uncertainty in kpc due to random and systematic errors is estimated to be about ± 10 percent. A significant reduction in this uncertainty should be possible by more precise measurement of the effective irradiance and by the use of the exact equation in place of the approximate one. The coefficient of variation of the flame spread factor computations was approximately 4 percent based on fifteen sets of four repeat determinations.

5. Conclusions

Inasmuch as the movement of hygroscopic moisture and the conduction of heat are basically diffusion processes, no abrupt difference in flame-spread behavior was expected with variations in the moisture content of typical combustible materials. The results did, in fact, indicate that the ignition sensitivity, or flame-spread factor, varied in a manner closely approximating previous data--for thick cellulosic materials conditioned at a single relative humidity--when the effect of moisture on the appropriate thermal properties was taken into account.

The radiometric method described for the direct and rapid in situ determination of the thermal inertia for surface heating (kpc product), is particularly attractive for building materials, including wood and other inhomogeneous materials. Although simple measurements are conveniently made at ordinary temperatures, the method may be readily applied to measurements at much higher temperatures. The application of the method to the rapid non-contact measurement of moisture content of materials also appears feasible. Finally, consideration could be given to the use of the method for applications involving composite materials with a thin surface veneer, for which the appropriate temperature-rise relationship (cf. ref. 11) should be employed.

6. References

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APPENDIX 1

The error in using the approximation $\sqrt{\frac{2b}{\pi}} = (1 - e^{-b^2} \operatorname{erfc} b)$ will be within 1, 5 and 10 percent for values of $b = 0.011$, 0.056 and 0.111, respectively. For a range in thermal inertia values, and assuming a room temperature value for H (the effective heat loss coefficient for radiation and convection) of $0.00084 \text{ w/cm}^2\text{C}$, these correspond to the maximum measurement times listed in Table 4.

Table 4: Error Introduced by Using the Approximation for Various $k\rho c$ Values and Maximum Measurement Times.

| Thermal Inertia $k\rho c$ | 1% ($b = 0.011$) | 5% ($b = 0.056$) | 10% ($b = 0.111$) |
|---|-----------------------|-----------------------|------------------------|
| $\frac{w^2 \text{ sec}}{^\circ\text{C}^2 \text{ cm}^2}$ | sec | sec | sec |
| 175×10^{-6} | .03 | .78 | 3.1 |
| 700×10^{-6} | .12 | 3.1 | 12. |
| 1750×10^{-6} | .3 | 7.8 | 31. |
| 7000×10^{-6} | 1.2 | 31. | 120. |

For very good insulating materials (low thermal inertia), increased accuracy may be achieved by continuing temperature-time observation for an extended period. A plot of temperature rise vs $1/\sqrt{t}$ becomes linear for long times and the value of $k\rho c$ may be determined from the slope of the line $\left(\frac{-I\sqrt{k\rho c/\pi}}{H^2} \right)$ and the intercept (I/H).

APPENDIX 2

The surface temperature rise of a semi-infinite solid subjected to a pulsed surface flux of rectangular wave form is equal to the sum of a periodic part superposed on the rising temperature due to the average surface flux fI . The value of the transient part is given by:

$$\theta_t = 2fI \sqrt{\frac{t}{\pi k \rho c}} \quad (8)$$

and the periodic part at time bT , after the beginning of a heating period [12], by:

$$\theta_p = 2fI \sqrt{\frac{T}{\pi k \rho c}} \left[(1 - f) - \frac{\phi(f, b)}{\sqrt{\pi b}} \right] \quad (9)$$

where T is the total time per cycle.

The ratio of the periodic to the transient parts is:

$$\left[(1 - f) - \frac{\phi(f, b)}{\sqrt{\pi b}} \right] \sqrt{\frac{T}{t}} \quad (10)$$

For $f = 0.5$, the average half-cycle value of the term in brackets is less than 0.24, so that for a chopping frequency of 90 cps ($T = 0.011$ sec), the effect becomes small ($<5\%$) for times greater than 0.25 sec.

Table 1.

Thermal Inertia and Surface Flammability of
Fiberboard and Hardboard

| Material | ρ g/cm ³ | Thickness cm | Relative Humidity percent | Moisture Content percent | w^2 sec/ $^{\circ}\text{C}^2$ cm ⁴ | $\frac{a}{\text{F}}$ kpc ⁻¹ sec/ $^{\circ}\text{C}^2$ cm ⁴ | $\frac{b}{\text{F}}$ s | $\frac{b}{\text{I}}$ s |
|---------------------------|-----------------------------|-----------------|---------------------------------|--------------------------------|--|---|---------------------------|---------------------------|
| Fiberboard (unpainted) | 0.27 | 1.19 | 0 | 0.8 | 2.5×10^{-4} | 26.3 | 26.3 | 500 |
| | 0.27 | 1.20 | 12 | 3.1 | 3.0 | 21.8 | 21.8 | 403 |
| | 0.28 | 1.22 | 50 | 6.9 | 3.7 | 18.5 | 18.5 | 328 |
| | 0.29 | 1.26 | 76 | 11.2 | 4.5 | 14.8 | 14.8 | 266 |
| | 0.31 | 1.30 | 99-100 | 25.4 | 6.3 | 11.0 | 11.0 | 146 |
| Fiberboard (painted) | 0.27 | 1.19 | 0 | 1.1 | 2.6 | 15.7 | 15.7 | 239 |
| | 0.27 | 1.20 | 12 | 3.7 | 3.1 | 13.6 | 13.6 | 191 |
| | 0.28 | 1.22 | 50 | 6.9 | 3.7 | 11.5 | 11.5 | 138 |
| | 0.29 | 1.26 | 76 | 11.2 | 4.5 | 8.82 | 8.82 | 96 |
| | 0.31 | 1.30 | 99-100 | 27.2 | 6.4 | 7.41 | 7.41 | 60 |
| Hardboard | 1.04 | 0.60 | 0 | 0.4 | 14 | 5.49 | 5.49 | 290 |
| | 1.04 | 0.60 | 12 | 1.9 | 16 | 4.94 | 4.94 | 244 |
| | 1.03 | 0.62 | 50 | 5.1 | 20 | 4.33 | 4.33 | 191 |
| | 1.02 | 0.65 | 76 | 9.0 | 24 | 4.05 | 4.05 | 181 |
| | 0.94 | 0.80 | 99-100 | 16.0 | 29 | 3.71 | 3.71 | 130 |

a/ Average for 2 specimens.b/ Average for 4 specimens.

Table 2.

Methods Used for Maintaining Various Relative Humidity
Conditions

| <u>Nominal Relative Humidity percent</u> | <u>Means of Achieving Humidity</u> |
|--|--|
| 0 | Calcium Chloride (CaCl_2) |
| 12 | Lithium Chloride ($\text{LiCl} \cdot \text{H}_2\text{O}$) solution |
| 50 | Commercial Room Air Conditioner |
| 76 | Sodium Chloride (NaCl) solution |
| 80 - 100 | Commercial Humidity Chamber |

Table 3

Effective Irradiance Measurements

| Material | Density | Thermal ^{a/} Conductivity | Heat Capacity | Thermal Inertia | Effective Irradiance |
|-----------------------------------|-----------------|---------------------------------------|--------------------------------|--|-------------------------|
| | ρ | k | c | $k\rho c$ | I |
| | g/cm^3 | $\text{w/cm}^\circ\text{C}$ | $\text{w sec/g}^\circ\text{C}$ | $\frac{\text{w}^2 \text{ sec}}{^\circ\text{C}^2 \text{ cm}^4}$ | w/cm^2 |
| Rubber | 0.97 | 15.2×10^{-4} | 1.77 | 26×10^{-4} | .020 |
| Insulating Board, dry | 0.23 | 5.03 | 1.21 | 1.4 | .019 |
| Rigid Poly- Vinyl Chloride | 1.4 | 12.5 | 0.84 | 15 | .018 |
| Asbestos Board | 0.67 | 12.3 | 0.84 | 6.9 | .016 |
| Gypsum- Vermiculite Plaster | 0.63 | 18.9 | 1.02 | 12 | .015 |

^{a/} Based on measurement in guarded hot plate apparatus (ASTM C117-42T)
at mean temperature of 23°C.

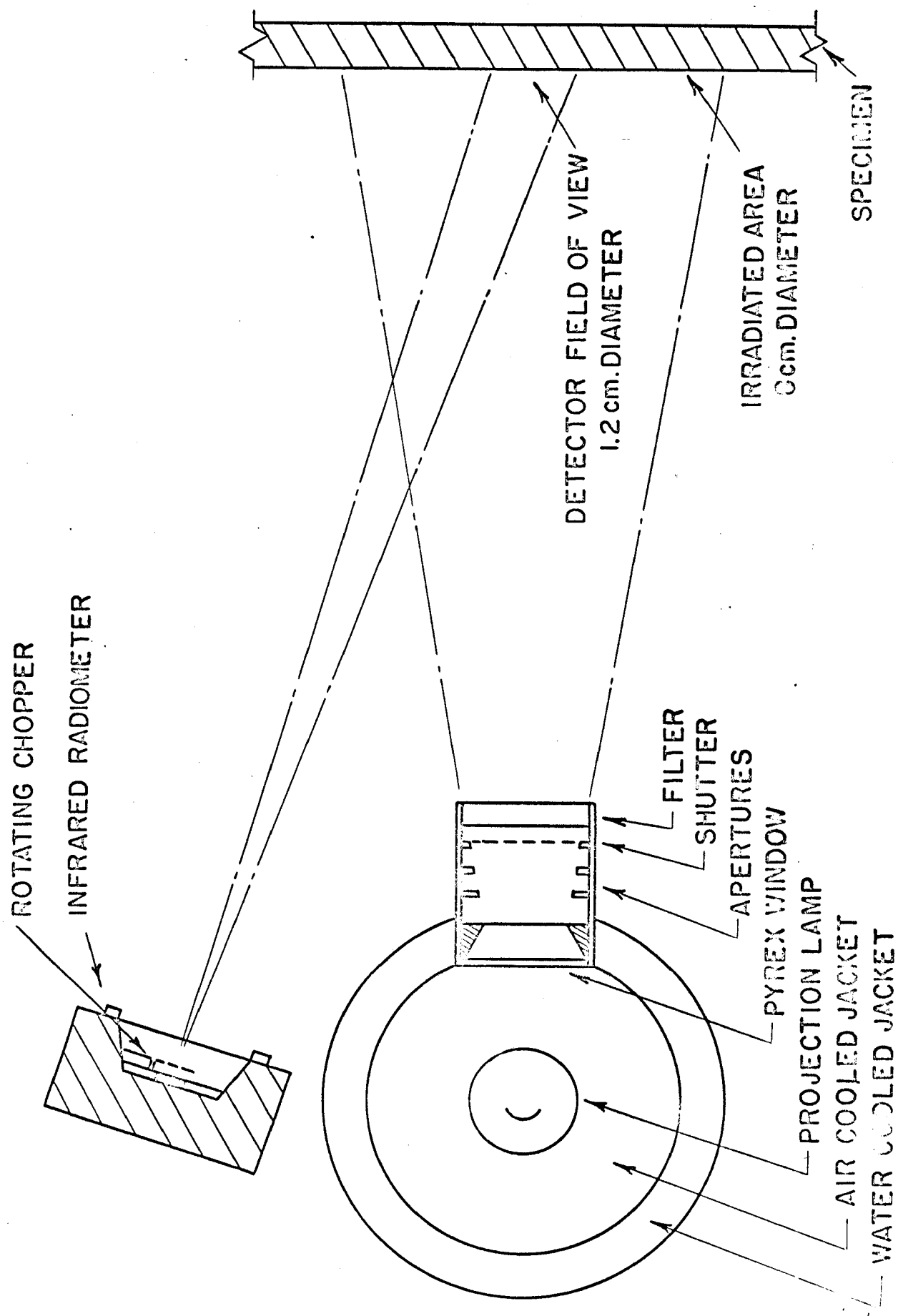


FIG.1-- SCHEMATIC DIAGRAM OF APPARATUS FOR $k\rho c$ MEASUREMENT

